A PROPOSAL FOR

Development Of A Polymeric-Nanoparticle Indoor Air Filter

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DEVELOPMENT OF A POLYMERIC-NANOPARTICLE INDOOR AIR FILTER

Proposed by Dr. Charlene W. Bayer, Georgia Tech Research Institute

INTRODUCTION

We are proposing to develop a prototype indoor air filter capable of controlling environmental tobacco smoke (ETS) odors and other airborne indoor contaminants, particularly in hotel rooms. The filter will fit into existing filter housings of HVAC units and will be designed to capture both particulate and gaseous contaminants. The first prototype will be designed to operate effectively for 30-60 days.

The removal of pollutants will be accomplished by air contact with an absorbent media -- a polymer or gel coated on a rigid support. In order to capture specific volatile pollutants and to improve the absorbency of the gel, nanoparticles may be added to the absorbent. Filter performance will be demonstrated through both mathematical models; environmental chamber testing using GTRI-developed, AHAM, and ASHRAE standard methods; and in a pilot field study.

RESEARCH TEAM

The research team will be lead by Dr. Charlene W. Bayer as the Principal Investigator. Dr. Daniel P. Campbell will lead the polymeric development portions of the project. Dr. Charles A. Eckert will lead the theoretical research. Mr. Robert J. Hendry will lead the chamber and field testing portions of the project.

TECHNICAL APPROACH

Three types of filters will be considered:

1. Polymer or gel coated on a rigid support,
2. Polymer or gel combined with nanoparticles coated on a rigid support, and
3. Filter system of nanoparticles.

The first two types of filters will be the primary focus of the research. The third only will be considered if the first two do not meet the project needs. The nanoparticles will be supplied by Nantek, Inc. through an agreement that Nantek has with Ecolab, Inc. The GTRI/GIT researchers will work directly and indirectly with the Nantek researchers under the auspices of Ecolab.

The research will be conducted in phases:

1. Theoretical and empirical testing of polymeric/gel materials for capture of targeted compounds,
2. Empirical testing of nanoparticles for capture of targeted compounds,
3. Theoretical and empirical testing of polymeric/gel materials combined with nanoparticles for capture of targeted compounds,
4. Small-scale chamber testing of selected polymeric/gel and polymeric/gel plus nanoparticle materials for capture of targeted compounds,
5. Design of filter system – coating of the selected polymeric/gel and polymeric/gel plus nanoparticle materials on rigid supports, such as honeycombs,
6. Testing of the filter system in the GTRI environmental chamber facility and in the ASHRAE 52.2 test rig,
7. Conducting a pilot field study by installing the filter in selected hotel field sites and monitoring of the removal efficiency over the lifetime of the filters.
8. Preparation of at least ten prototype filters for Ecolab, and
9. Working with Ecolab to set up manufacturing of the filters.

In the initial phase, polymeric/gel materials will be tested theoretically and empirically for capture efficiency of targeted compounds, including formaldehyde, carbon monoxide, ammonia, nicotine, hexane, toluene, and ETS particles. Additional compounds will be selected based on known ETS airborne contaminants and the availability to obtain sufficient quantities of the standard compounds and capability to aerosolize a standard amount of the compound. For example 3-ethenylpyridine is a marker compound for ETS in air, but is not available in large enough quantities to useful as an aerosolized standard for testing during the initial phases of this project. This compound will be useful to track during the environmental chamber and field study phases of this project. The ETS target compounds developed by the State of California will be considered as standard analytes.

Similar testing will be done on the polymeric/gel plus nanoparticles. The nanoparticles may interfere with some of the theoretical and interferometric testing since the optical path may no longer be clean. If this occurs, the polymeric/gel plus nanoparticles will be tested for capture efficiency by placing in very small lab-bench scale chambers for testing with spiked airflows. The concentrations will be measured up- and downstream to determine removal efficiency. This system also will be used to test the nanoparticle removal efficiencies.

FILTER SYSTEM DESIGN

The filtration system design requires detailed knowledge of the phase equilibria and transport properties of the air-pollutant-absorbent system. To avoid the prohibitive expense and time delay of constructing and testing a large number of prototype filters, filter characteristics will be determined and optimized by established transport and phase equilibria calculations. Solving all calculations simultaneously may require

development of a computer program using finite element analysis. The design calculations include the following:

- Transport of pollutants from the air to the absorbent surface
  - ✓ These calculations allow for the estimation of filter parameters such as the airflow configuration and the required surface area of polymer absorbent.

- Transport of pollutants from the absorbent surface to the gel interior
  - ✓ These calculations allow for the optimization of filter parameters such as the absorbent coating thickness, and the length of filter required.

- Phase Equilibria
  - ✓ These calculations will be used to estimate required inputs into the transport calculations such as the pollutant partitioning between air and the absorbent, and the maximum loading of pollutants in the absorbent.

- Economics
  - ✓ These calculations will be used to estimate and optimize filter production costs with parameters that determine filter performance.

- Material Balances
  - ✓ These calculations allow for the estimation of filter parameters such as the minimum mass of coating required and will provide necessary inputs for the economic calculations

The theory used to calculate mass transfer and phase equilibria is fairly well established for the design constraints of this project, and calculations will greatly reduce the number of experimental tests required. However, transport and phase equilibria properties for specific pollutants and absorbents can be estimated only roughly; they must be measured experimentally to ensure accurate filtration system design.

THEORETICAL CALCULATION EXPERIMENTS

The transfer of a given pollutant from air into an absorbent is primarily a function of the physical configuration of the filter, temperature, airflow rate, pollutant concentration in the air, diffusivity of the pollutant in air, diffusivity of the pollutant in the absorbent, and the thermodynamic partitioning of the pollutants between air and the absorbent.

The temperature, airflow rate, and pollutant concentration in the air are easily determined, and the diffusivity of the pollutants in air at ambient conditions can be reasonably estimated. The diffusivity of the pollutants in various absorbent phases, and the partitioning of the pollutants between the air and the absorbent, however, is not easily estimated with sufficient accuracy for this filtration system design.

We currently have the expertise and equipment to measure these properties as well as the ability to evaluate prototype filter performance by standard AHAM and ASHRAE methods. While the transport and phase equilibria properties of all pollutants will not be measured in the duration of this project, a sufficient number and variety of model compounds will be used to estimate a much wider variety of pollutants.

POLLUTANT PARTITIONING BETWEEN AIR AND ABSORBENTS

The thermodynamic partitioning of the pollutants between air and the absorbent will be determined by several experimental methods already established in our laboratories. A uniform absorbent film of known thickness is placed in contact with air containing a known concentration of pollutants. The uptake of the pollutant by the absorbent film is then measured by interferometry or by UV and IR spectroscopy after the system has come to equilibrium.

UV/IR SPECTROSCOPIC METHOD. A sealed vessel with two connected optical paths is cleaned to remove all contaminants. A film of absorbent is placed in one of the optical paths (Figure 1) and baseline spectra are taken for both optical paths. Air with a known concentration of pollutant is then introduced into the vessel and the system is allowed to equilibrate. The pollutant uptake of the absorbent can be determined by subtracting the spectral absorbance of the empty optical path from the spectral absorbance of the optical path containing the absorbent film.

Figure 1. Schematic of parallel path cell.

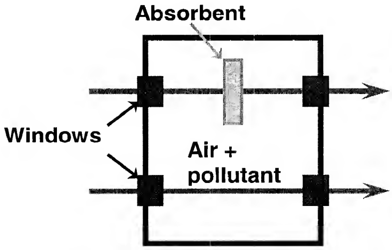
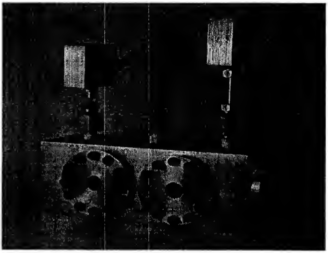
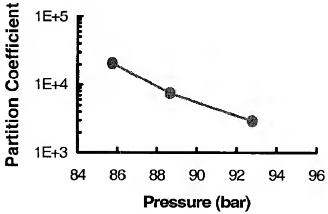


Figure 2. Photograph of parallel path cell



Depending on which pollutants are used, either UV-vis or IR spectroscopy may be used for this determination. Absorbent films with known loadings of pollutants may be required for calibration of these measurement techniques. Data previously measured in Dr. Eckert's laboratories are shown in Figure 3.

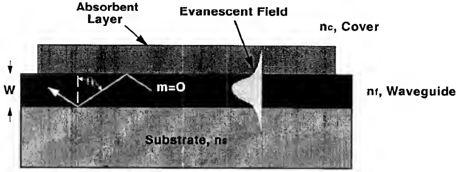
Figure 3. Partitioning of an azo dye (diethylaminonitroazobenzene) between poly(methyl methacrylate) and carbon dioxide at 40 °C with various pressures of carbon dioxide.



Planar Waveguides and Interferometry. The partitioning of pollutants between air and an absorbent may also be measured by interferometry. A film of absorbent is cast on the surface of an optically transparent planar waveguide. Planar

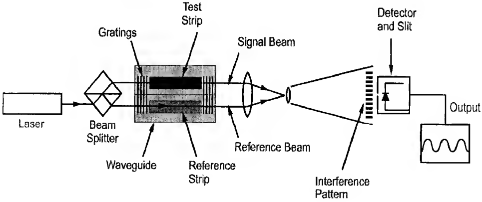
waveguides have evanescent fields sensitive to index of refraction changes in the volume immediately above the waveguide surface. These fields extend up to 5000 Å above the surface (Figure 4). Placing an absorbent film within this field allows one to monitor changes that take place within this film. Chemical or physical interactions

Figure 4. Diagram for single mode waveguide with buried evanescent field



change the index of refraction causing the propagating light speed, or phase, to change in a direction opposite to that of the index change. To measure this change a reference-propagating beam, adjacent to the sensing beam, is optically combined with the sensing beam creating an interference pattern of alternating dark and light fringes (Figure 5). When chemical or physical changes occur in the sensing arm the interference pattern will shift producing a sinusoidal output. With detectable sensitivities changes as small as

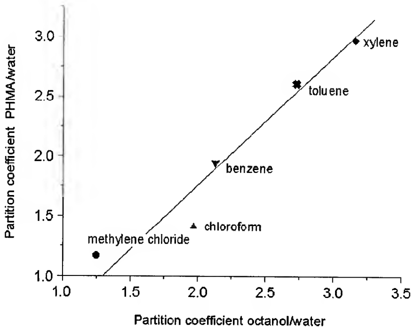
Figure 5. Diagram of waveguide interferometer setup



10^{-6} can be measured. The resulting phase shift is a direct measure of the change in refractive index due to the uptake of pollutant by the absorbent. This refractive index

change can be correlated with the bulk concentration of pollutant in the absorbent. The ratio of pollutant in the airflow and in the adsorbent film is a measure of the equilibria or partition coefficient. Figure 6 shows the partition coefficient between poly(n-hexyl methacrylate) and water measured by planar waveguide interferometry versus published values for the octanol/water partition coefficient for several analytes. Although these data were obtained with in an aqueous experiment the same information can be obtained in the vapor phase.

Figure 6. Partition coefficient of poly(n-hexyl methacrylate)/water versus octanol/water.



The rate of phase change can be used to determine the diffusion of the pollutant in the absorbent. In addition, varying the absorbent film thickness will allow differentiation between surface adsorption and bulk absorption. This differentiation may be necessary for filtration system design and scale-up. This method may also require calibration with absorbent films containing known concentrations of pollutants and depends on a difference in refractive index between pollutants and absorbents.

Diffusivity of Pollutants into Absorbents

The diffusivity of the pollutants into the absorbent will be measured through a similar experiment by using a thicker absorbent film and measuring the uptake of

pollutant by the absorbent film with time. A second technique may be employed where the transfer of pollutants through a thin film of absorbent is measured with time by spectroscopy or by interferometry. Diffusivities measured by this technique in our laboratories are shown in Figures 7,8 and 9. Figure 7 shows the diffusion rate for the polymer poly(2,2-bistrifluoromethyl-4,5-difluoro-1,3-dioxole-*co*-tetrafluoroethylene) for several different analytes as measured by interferometry. Figure 8 shown the diffusion of methylene chloride into a series of different polymers also measured by interferometry. Though both the data used in Figure 7 and 8 were obtained in the aqueous phase similar measurements can be obtained in the vapor phase. Note that in some cases the diffusion is linear with concentration and in other cases it appears to increase with concentration indicating some synergistic effect. Figure 9 shows the diffusion of diethylaminonitroazobenzene in poly(methyl methacrylate) in the vapor phase.

Figure 7. Diffusion Rates for various analytes in poly(2,2-bistrifluoromethyl-4,5-difluoro-1,3-dioxole-*co*-tetrafluoroethylene)

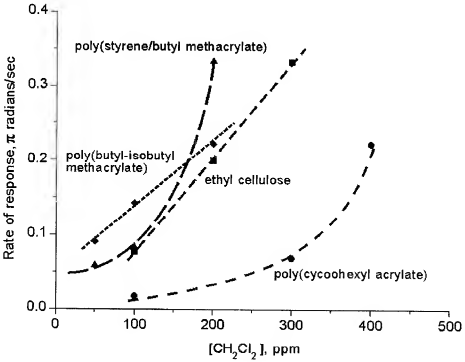


Figure 8. Diffusion of methylene chloride in various polymers

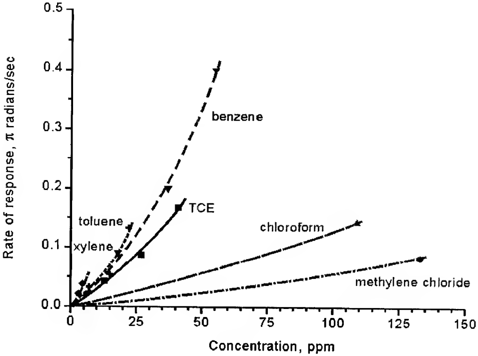
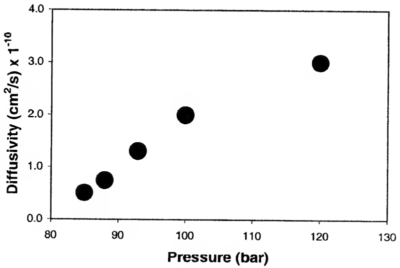


Figure 9. Diffusivity of an azo disperse dye, diethylaminonitroazobenzene, in poly(methyl methacrylate)



ABSORBENT SELECTION

Design of a successful filter will require the screening and selection of the proper absorbent or mixture of absorbents and may require the use of nanoparticles to meet design requirements. Absorbents will be selected and screened based on the following criteria:

- Cost
- Pollutant partitioning
- Viscosity/diffusivity of pollutants in absorbent
- Resistance to flow/bleed
- Ease of coating and solvent removal from coating
- Tackiness for particulate capture
- Compatibility with humid air
- Environmental impact—filter coating method as well as disposal and recycling

A filter capable of efficiently capturing a wide variety of pollutants may include a composite of several different absorbents in series. Compounds with reactive functionality may be sequestered by absorbents with complementary reactive functionality. For example, pollutants with acid functionality may be sequestered by polymers with amine functionality.

Polymer choice involves looking at the four corners of the matrix of polymers derived from hydrophoblic polymers and hydrophilic (such as the hydrogel), low glass transition polymers and high glass transition polymers. The low glass transition polymers will slow diffusion versus the high glass transition polymers due to the lack of void volume. However, slow diffusion out may be quite advantageous for pollutant retention. Nanoparticles will be examined with polymers that possess the desired properties for pollutant adsorption and if pollutant sequestering is achieved the use of high tg polymer with faster diffusion may prove to be the best at pollutant capture. Screening of these polymers will be done using the interferometric setup and the data analyzed theoretically by the chemical engineers. Their results will direct the direction of subsequent polymer choice.

Initial choice include the N-methyl acrylamide-*co*-acrylamide hydrogel, polyisobutylene, Teflon AF™ and poly(vinyl alcohol). Additional polymers with acid and base functionalities will be looked at for sequestering of acid/base pollutants versus the nanoparticle containing polymers. Polymers to start with can be polyacrylic acid and polyethyleneimine.

COATING OF SUPPORT AND FABRICATION OF PROTOTYPE FILTRATION SYSTEMS

Coating of planar waveguide substrates for polymer evaluation will be done by dip coating or spin coating. Thickness of the deposited polymers will be done using polfilometry, which is accurate to 10's of angstroms. Coating of test filters will be done

by dip-coating. Thicknesses in both cases are controlled by concentration of polymer, solvent and dipping speed or spin rate.

Choices of filter substrates initially will be either of a honeycomb design or a Venetian blind, singular or multiple, configuration. The final selection will be based on ease of coating, effect on capture efficiency and system backpressure, cost, and manufacturability. Ecolab will be consulted on these issues, particularly the costs and manufacturability. Mr. H.E. Burroughs, an internationally recognized filtration expert, may be consulted at this stage on manufacturability issues.

Once the filter design issues have been resolved the filters, prototype filters will be made for testing in the GTRI environmental chamber facility and in the GTRI ASHRAE 52.2 test rig.

FILTER TESTING

The prototype filters initially will be tested by installing them in the GTRI 28.3 m^3 environmental chamber. The filters will be tested for capture efficiency against ETS, generated by a smoking machine, and authentic standards following a modified AHAM testing method. The final authentic standards for the test will be determined during the earlier phases of the project, but will include at a minimum ETS (both particulate and gaseous phases), formaldehyde, carbon monoxide, and toluene. The filters will be tested to determine lifetime, capture efficiency, and potential re-emission of captured pollutants. Based on this testing if modifications in the filter are required, then the prototypes will be redesigned based on additional laboratory work and retested.

Once the prototype filters met the criteria required for this stage of testing, they will be taken to the GTRI ASHRAE 52.2 test rig and tested according to ASHRAE Standard 52.2 for particulate contaminants removal and according to GTRI research methods for gaseous contaminants removal. Based on this testing if modifications in the filter are required, then the prototypes will be redesigned based on additional laboratory work and retested.

PILOT FIELD TEST

At least ten prototype filters will be made for use in a pilot field test. GTRI researchers will work with Ecolab researchers to select a hotel site for the field test. The field site will be monitored for indoor air contaminants, particularly ETS, prior to installation of the prototype filters to determine the background levels. The filters then will be installed and the indoor air quality will be monitored for approximately 60 days, monitoring both rooms with and without the filters installed. The improvement in the indoor air quality created by the filters will be determined.

PHASE II WHITE PAPER

BAA NUMBER: N-39998-00-Q-0808

MISSION AREA: Chemical, Biological, Radiological, and Nuclear Countermeasures

REQUIREMENT NUMBER: R-641 Chemical and Biological Adsorption Filter Technology (CB-R-641-GTRI-001)

PROPOSAL TITLE: Advanced Adsorption/Particle Filtration System for Protection Against Chemical/Biological Threat Agents

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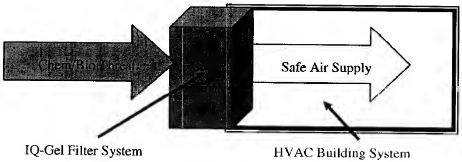
Advanced Adsorption/Particle Filtration System for Protection Against Chemical Biological Threat Agents



There is growing concern among civilian and military communities about the potential of terrorist attacks with chemical and biological (CB) warfare agents. The most indiscriminate delivery method of such agents is via an aerosol attack so that the agents are transported to the objective as gases or aerosol mists, in which particle or droplet sizes are in the respirable size range (≤0.6 μm), and so that a sufficient does of the CB agent is transmitted to cause sickness and/or death. A particularly vulnerable area for attack is building ventilation (HVAC) systems at the outdoor air intake. An HVAC system is designed to draw in large amounts of outside air, mix it with a percentage of air already in the building, and transport that mixed air throughout the building, particularly to occupied zones. The ventilation system is the air pathway throughout a building. Release of a CB agent near a building's outside air intakes will allow distribution of the agent throughout the building, unless some type of filtration/air cleaning system exists in the ventilation system that captures the agent preventing agent transference into the building air and ventilation system. *We are proposing to develop this type of filtration/air cleaning system.* Any building is vulnerable to this type of attack with a CB agent or other types of toxic/hazardous, more common and readily available chemicals (such as phosgene, chlorine, hydrogen sulfide, methyl mercaptan, or arsine). US embassies and military buildings potentially are targets of this type of threat.

Technology Outline

Proposed Solution: We are proposing to develop an enhanced filtration/air-cleaning/capture system that will remove and trap airborne chemicals and particles from an airstream. Our proposed system will use the GTRI- patented IQ-Gel (US Patent 5,529,609), removing gaseous agents by adsorption/absorption and particulate

agents (including biological agents) by adhesion, and a GTRI-patented integrated optic sensor (U.S. Patent 4,940,328) indicating the presence of a threat agent and filter exhaustion. The IQ-Gel will be formulated so it that can be placed into standard filter housings replacing conventional filters in commercial building HVAC systems, thus easily retrofitted to existing facilities. Three different formulation avenues will be pursued: (1) applying the gel to a polymeric screen mesh, a screen made of the IQ-Gel, or a commercially available filter, (2) constructing of a fiber bed of the gel, and (3) forming gel pellets or coating of the gel on polymeric beads. Interferometric sensors will be imbedded in the filter system at multiple points to indicate the progression of a threat agent through the filter system bed to prevent exhaustion of the filter system and breakthrough of the agent into the building supply air. The optimal criteria to be met by the IQ-Gel filter system are: 2'x2'x12' filter size, pressure drop across the filter system $\leq$3.0 iwg, pressure drop across each individual filter section $\leq$1.5 iwg, particle removal efficiency >99.97% for particle sizes between 0.3-10 μm, adsorption efficiency >99% for nerve, blister, and blood agents from ambient concentration of 5X IDLH with a breakthrough time of $\geq$15 minutes, and broad adsorption spectrum for acid-forming and basic toxic chemicals. Specific goals to be pursued are: a filter lifetime of three years, low cost, regeneration of the filter rather than disposal, and the development of a sensor for filter lifetime indication.

POTENTIAL USERS: The proposed technology has dual use capabilities for both military and civilian applications. Potentially it could be used in any building HVAC system, since it should remove common airborne pollutants, as well as CB agents. US Embassy buildings, military facilities, commercial buildings, and personal residences are potential for use areas for the proposed filtration/air-cleaning/capture system. The proposed system should replace the currently used particle filters in the building HVAC systems. The sensor as a filter lifetime indicator will have many applications beyond combining with the IQ-gel filter. Currently such an indicator of filter life, especially for gases removal, does not exist.

THE IQ-GEL: The IQ-Gel matrix consists of an acrylamide polymerization with the cross-linking agent, n-methylol acrylamide, in the presence of a plasticizing agent, glycerol. The IQ-Gel's properties of tack, viscosity, tensile strength, and pliability can be adjusted by varying the ratios of the ingredients and by varying reaction time between the ingredients, which will allow the formulation of the gel into the proposed three filter forms. Particles adhere to the media for removal from the air stream, and after adhesion move into the interior of the gel mat. The adhesion is dependent on surface energy rather than particle size, therefore the IQ-Gel will remove large and small particles, including living organisms – dust mites, bacteria, viruses, and fungi. Once a particle has adhered, it is not

released back into the air stream. Gaseous pollutants are removed by a combination of adsorption and absorption. The non-optimized prototype filter, developed at GTRI during previous research projects, has demonstrated a capacity ratio for volatile organic compounds (VOCs) greater than seven times its weight, based on challenge mass and loading weight of challenge gas mixtures of authentic standards. This is capacity is significantly greater than most other known adsorbents. No re-emission was detected. Additionally the IQ-Gel does not support microbiological growth, since it does not have sufficient water activity to allow for proliferation and amplification of the organisms. Additional materials can be added to the IQ-Gel to augment its performance, including dyes, absorbents or strengthening material. Adsorbents, like Zeolites, and reactive compounds, such as metal catalysts, can be incorporated to tailor its removal abilities to specific applications. The IQ-Gel is the only known air cleaning media that can remove gases, particles, and microbes in a single pass on a single media.

PREVIOUS WORK: The IQ-Gel prototype development was funded under two projects: (1) by SC Johnson and Son and GTRI (total funding $50,000) resulting in the GTRI patent "Air Cleaner Having a Three Dimensional Visco-Elastic Matrix of Material" (Patent #5,529,609) (report submitted to SC Johnson and Sons 1995), and the Advance Technology Development Center (ATDC) under the Faculty Research Commerialization Program (total funding $54,000) (report submitted to ATDC 1998). From this work, prototypes of IQ-Gel filter system have removed 99% of respirable-sized particles and greater than 90% of organic compounds (Table 1). The drawback to commercialization so far has been formulating the IQ-Gel into a form with sufficient inflexibility to be used in an HVAC system. The two previous projects were not sufficiently funded to be able to carry out this task..

Table 1. VOC reduction % for challenge gas mixture

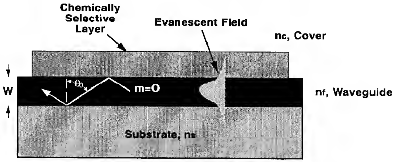
Day #	% Reduction					
	Methylene Chloride	Ethyl Acetate	Methyl Ethyl Ketone	Trichloro-ethane	Hexane	Total Volatile Organic Compounds
1	62	47	59	40	79	59
2	85	55	74	44	81	73
4	95	70	84	86	90	92
5	97	80	80	85	93	93

Under the first research project, the IQ-Gel was made and evaluated in a small stand-alone air cleaner installed in a 28.3 m^3 environmental chamber and challenge tested with gas mixtures of the compounds in Table 1 (class representatives of common indoor air pollutants) and ambient particles. The advantages of incorporation of adsorbents, such as Zeolite A, for enhanced VOC adsorption were investigated. Although enhanced removal efficiencies were obtained, the gel without added adsorbents performed sufficiently well for the project goals project without the added

expense of the additional adsorbents. During the ATDC funded project, the gel was formulated into a latex that was sprayed onto metal grease filters and residential fiberglass furnace filters, at the request of our industrial partner Semco Inc. Using these two matrices for support of the gel did not allow for a sufficient amount of the gel to be coated on the matrix for lifetimes greater than a few days. The gel also was formulated into several different forms, by varying the reaction conditions and ingredient amounts, ranging from a pourable liquid to a diceable hard material. IQ-Gel-coated metal grease filters installed in place of high efficiency filters in the HVAC system of a Florida yacht club for 48 hours. During this time, occupants remarked about how clean and pure the air felt. Measurement of contaminants in the indoor air with the IQ-Gel filters in place indicating some improvement in the indoor air quality. The gel-coated filters also were installed in the filtration system of the GTRI 28.3 m^3 environmental chamber and challenged and lifetime tested under controlled conditions. We determined that the IQ-Gel captures greater than seven times its weight in airborne pollutants, but the lifetime was only a few days due to the small amount of material that could be sprayed onto the support media without damaging the support media. We hypothesized that IQ-Gel formulation should be in a pellet or coated bed, a fiberbed, or coated on a media with matched surface energies. Dr. Gooch, one of the co-inventors, has demonstrated that the IQ-Gel can be extruded into fibers and formed into pellets.

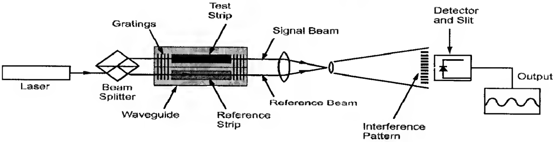
INTERFEROMETRIC SENSOR: In conjunction with the IQ-Gel filter system, an indicator/lifetime/breakthrough sensor system will be developed, which will be imbedded in several points of the filter media to measure the progression of a threat agent into the filter media. The sensors will detect potential failure or actual failure of the filtering ability of the IQ-Gel filter system. The sensor should be able to determine the identity of the breakthrough analyte and provide a warning to either change the filter, shut down the filtering system or evacuate the building. This sensor will be based on the GTRI patented interferometric sensor, which has been used for sensitive detection and quantitation of a number of chemical and biological species. This sensor consists of an optical planar waveguide interferometer, which contains thirteen sensing regions on a single miniature platform. Planar waveguides have evanescent fields sensitive to index of refraction changes in the volume immediately above the waveguide surface. These fields extend up to 5000 Å above the surface. Placing a chemically sensitive film within this region provides the basis for a chemical sensor (Figure 1). Chemical or physical interactions change the index of refraction causing the propagating light speed, or phase, to change in a direction opposite to that of the index change. To measure this change a reference-propagating beam, adjacent to the sensing beam, is optically

Figure 1. Diagram for single mode waveguide with buried evanescent field



combined with the sensing beam creating an interference pattern of alternating dark and light fringes (Figure 2).

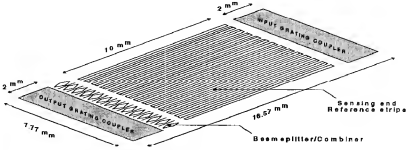
Figure 2. Diagram of non-integrated waveguide interferometer setup



When chemical or physical changes occur in the sensing beam, the interference pattern will shift producing a sinusoidal output. With detectable sensitivities on the order of 0.02 π radians, index changes less than 10^{-6} can be measured.

Tailored chemistries applied to the waveguide can be passive (e.g.; inducing swelling or dissolution in a film) or active (e.g.; containing reactive or binding sites). Fast and reversible chemistries usually are the goal, in most cases, for both gaseous and liquid applications; however, an integrating chemistry can just as easily be designed to determine very low concentrations of a given analyte such as chemical warfare agents. Passive mechanisms are used when the target analyte is relatively inert, *i.e.* aromatic and chlorinated hydrocarbons. Active chemistries developed include tailoring the acid-base strength of the sensing film, the nucleophilicity or electrophilicity of the film, and antibody-antigen binding. Sensitivities range from the low ppm to low ppb for vapor and aqueous applications, and 0.01 pH units or ng/mL for biologicals. The currently developed sensor platform has 13 interferometers on a 1x2-cm chip (Figure 3). All components needed to launch the optical beam, interfere the optical waves and couple out the interference pattern are fabricated into the waveguide chip. A laser illuminates all channels across the width of the input grating. A patterned layer of thick SiO_2 defines the channel

Figure 3. Integrated chip with 13 interferometers, gratings and optics



lengths. In the case of the passive organic solvent sensor, coarse, medium and fine channels allow for increased sensitivity and dynamic range. Four polymers (three using the dissolution mechanism and one polymer using free volume filling) are used to provide a patterned output for analyte identification and concentration determination. In the active sensor, coarse and fine channels have been used in addition to separate channels for canceling possible interferants. Field testable units have been assembled with onboard electronics capable of converting interferometric signals to total phase shift, and reporting concentration and identification of single analytes and mixtures through pattern recognition and specific reactions.

PREVIOUS WORK: The sensor is being applied to a number of environmental, industrial, and packaging detection problems for a number of government (military and civilian) and industrial sponsors. Several of the applications to which the GTRI sensor is being applied and/or developed are: BETX sensing in water, humidity measurement, ammonia sensing during fertilizer application, airborne influenza virus, *salmonella* on processed poultry, *E-coli* detection, toxic industrial chemicals, and chemical warfare agents.

PROJECT OUTLINE

Task 1: *Gather data on targeted chemical/biological threat agents and other toxic/hazardous chemical agents that could pose a threat and develop a list of surrogate gaseous and particulate test materials. Evaluate targeted chemical/biological threat agents for adsorption/filtration requirements to be meet for required efficiency specifications.* Many candidate threat agents have similar structures and properties to readily available, less toxic industrial chemicals. For example many nerve agent chemicals are organophosphate based, the same chemistry as used for many pesticides; therefore organophosphate pesticides may be substituted as a surrogate test material for nerve agents. During this task, threat agents of interest will be classified and appropriate surrogate, less toxic chemicals (volatile, semi-volatile, and particulate) will be selected for challenge testing of the IQ-Gel filter to determine efficiency, performance, and lifetime. Information on the chemical and biological agents of interest need to be provided by the government.

Task 2: *Formulate GTRI-patented IQ-Gel into the three forms, validate its performance, and optimize for maximum efficiency and lifetime for targeted applications.* The criteria for filter formulation evaluation are: 1) sufficient airflow through the filter, 2) pressure drop across filter system, 3) stability of gel on support matrix, 4) removal and capture efficiency for targeted particles and aerosols, gases, and microbes 5) filter capacity, 6) filter lifetime, and 7) potential for commercialization. A critical point for making an IQ-Gel filter with sufficient quantities of gel to reach desired lifetimes is surface matching of the gel with the appropriate support matrix, and for the surface matrix to have enough rigidity to hold the weight of the gel. An approach to this problem is the selection of a polymeric screen with the appropriate surface characteristics. Dr. Campbell will investigate this approach, since this is a problem that he solved in the development of waveguides for the interferometric sensors. The proposed filter system probably will be used in conjunction with an existing pre-filter in the HVAC system to remove the larger particles to increase the gel filter lifetime. It is predicted that the three formulations will be made and preliminarily tested. The formulation that best meets the performance criteria will be selected to continue with validation with the surrogate compounds selected in Task 1. An appropriate filter housing system will be designed meeting the criteria that the filter system replace currently used HVAC filters. (This will be done with Mr. H.E. Burroughs, PE of Building Wellness Consultancy, an internationally recognized expert in filtration and indoor air quality). Validation will be with modified ASHRAE 52.2 Standard test method and using the method specified test rig modified for gaseous compound challenge testing, as well as particle efficiency testing. The particulate removal and capture efficiency will be measured by challenging the gel-filter with a range of dry KCl particles sizes. (ASHRAE 52.2 uses a particle size range of 0.30 to 10.00 μm, but this will be extended to 0.10 to 500 μm for this project.) The removal efficiency is calculated by measuring the concentration differences up- and downstream of the test filter. Organic (volatile and semi-volatile) compounds of interest will be injected into the modified ASHRAE 52.2 test rig and the removal efficiency calculated by measuring the difference in concentrations up- and downstream of the test filter. Lifetime will be evaluated by challenging the gel-filter with extremely high concentrations of target compounds to measure the capacity of the IQ-Gel for these substances, both gaseous and particulate. The lifetime will be predicted from these data, similar to ASHRAE Draft Standard SPC145. GTRI is currently funded by ASHRAE for a research project on gaseous filtration efficiency and service life determination that will provide input into the filter testing methods proposed for this project.

Materials incorporation into the IQ-Gel will be investigated to obtain greater lifetime, overall removal/capture efficiency, and/or specificity of removal/capture efficiency for target species. These materials may

include adsorbents, such as Zeolites, metal salts, or other reactive chemicals. Reactive chemical incorporation may be particularly useful for the capture and degradation of nerve and blister agents of acid-forming chemicals.

Task 3: *Develop lifetime indicator sensor. Incorporate into filter system and validate performance with surrogate compounds.* The interferometric sensor will be developed for the selected targeted compounds. It is anticipated that the sensor chemistry will be developed to measure compound classes rather than specific compounds to increase the likelihood of detection. Particles will be detected by changes in optical density of the surface.

Task 4: *Validate complete system in GTRI 28.3 m^3 environmental chamber system* with surrogate compounds and particles. Installation of the IQ-Gel filter in the HVAC system of the chamber will allow us to simulate its use in a building. The effects of changing temperature and humidity and challenge mixtures on performance also will be determined using the testing procedure described above. The performance under changing environmental conditions and differing pollutant loadings is a critical performance criteria. We predict that the IQ-Gel filter will perform best under higher relative humidity conditions, because the prototypes appeared to be very dry (no longer moist) at failure. Lifecycle calculations will be based on the cost of materials and construction, the efficacy and efficiency, any increased energy costs or equipment costs for operation, and potential costs for disposal or renewal (if possible).

Task 5: *Send to Aberdeen, or appropriate federal testing facility, for validation with actual threat agents.* GTRI does not have the facilities to test the gel-filter with actual threat agents. The gel-filter will have to be sent a governmental testing facility, such as Aberdeen, to measure performance with actual threat agents. The government will have to help select the testing facility and help arrange the testing and target threat agents for the challenge.

Task 6: *Develop description and drawings of adsorption/particle filter system in typical building system, specifications, model to predict system performance in buildings, and users' manual.* The final designs and report on performance, installation, performance, etc. will be prepared both on electronic and hard media. Dr. Kaiss Al-Ahmady of Indoor Air Solutions, a recognized expert in a indoor air modeling.

PERSONNEL

Dr. Charlene W. Bayer is a Principal Research Scientist and Branch Head at GTRI, Adjunct Professor in the School of Chemical Engineering and School of Earth and Atmospheric Sciences with the Georgia Institute of Technology, and a GTRI Fellow. Dr. Bayer has over 20 years of environmental analytical chemistry experience, specializing in separations sciences and mass spectrometry of environmental samples, particularly of air quality analysis. Areas of expertise involve development of new methods and use of current methods for organic compound

PHASE III PROPOSAL

BAA NUMBER: N-39998-00-Q-0808

MISSION AREA: Chemical, Biological, Radiological, and Nuclear Countermeasures

REQUIREMENT NUMBER: R-641 Chemical and Biological Adsorption Filter Technology (CB-R-641-GTRI-001)

PROPOSAL TITLE: Advanced Adsorption/Particle Filtration System for Protection Against Chemical/Biological Threat Agents

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DATE: October 6, 2000

Advanced Adsorption/Particle Filtration System for Protection Against Chemical Biological Threat Agents

Abstract:

An aerosol attack is the most indiscriminant delivery method of chemical and biological (CB) agents so that the agents are transported to the objective as gases or aerosol mists. A particularly vulnerable area is the building ventilation (HVAC) system, especially at the outdoor air intake, since the HVAC system is the air pathway throughout a building. A CB agent released near a building's outside air intake will distribute agent throughout the building, unless some type of filtration/air cleaning system is a component of the HVAC system capturing the agent preventing transference of the agent into the building air and ventilation system. *This proposed 19-month project will develop an enhanced filtration/air cleaning system that will capture CB agents preventing their transport into a building through the ventilation system air pathway.* The proposed filtration/air cleaning system will use the GTRI-patented IQ-Gel (US Patent 5,529,609) to remove and trap airborne chemicals and particles from the airstream. The IQ-Gel will be formulated so that it can be placed into standard filter housings replacing conventional filters in commercial building HVAC systems. Three different formulations will be investigated for optimal capture efficiency: (1) gel application to a polymeric screen mesh, a screen made of the IQ-Gel, a commercially available filter, or a carbon cloth, such as ASZM-TEDA; (2) construction of a fiber bed of the gel; and (3) forming gel pellets or coating the gel on polymeric or carbon beads. An optional task will develop an imbedded interferometric sensor based on the GTRI-patented sensor (US patent 4,940,328) as a lifetime indicator. Combination of the IQ-Gel filter with currently available technology such as ASZM-TEDA activated charcoal and HEPA particulate filtration will be investigated. Specific goals that will be pursued are: a filter lifetime of three years, low cost, filter regeneration, and method for filter lifetime indication. A total of 28 person-months, employing nine researchers is proposed, not including Option 1.

The proposed technology has dual use capabilities for both military and civilian applications. Potentially it could be used in any building HVAC system, since it should remove common airborne pollutants, as well as CB agents. The proposed system should replace the currently used particle filters in a building HVAC system.

ADVANCED ADSORPTION/PARTICLE FILTRATION SYSTEM FOR PROTECTION AGAINST CHEMICAL BIOLOGICAL THREAT AGENTS

TECHNICAL SECTION:

EXECUTIVE SUMMARY:

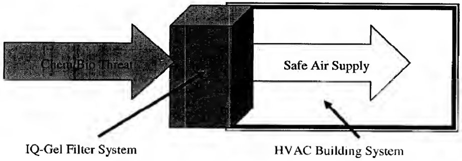
There is growing concern among civilian and military communities about the potential of terrorist attacks with chemical and biological (CB) warfare agents. If a CB agent is released near a building's outside air intakes, it will be distributed throughout occupied areas of the building, unless some type of filtration/air cleaning system exists in the ventilation system that captures the agent preventing agent transference into the building air and ventilation system. *We are proposing to develop this type of filtration/air cleaning system.* The proposed technology has dual use capabilities for both military and civilian applications. Potentially it could be used in any building HVAC system, since it should remove common airborne pollutants, as well as CB agents. The proposed system should replace the currently used particle filters in a building HVAC system.

We are proposing to develop an enhanced filtration/air-cleaning/capture system that will remove and trap airborne chemicals and particles from an air stream. Our proposed system will use the GTRI-patented IQ-Gel (US Patent 5,529,609), removing gaseous agents by adsorption/absorption and particulate agents (including biological agents) by adhesion, and a sensor or optical system will be incorporated to indicate the presence of filter exhaustion. The IQ-Gel system will be installed in standard HVAC filter housings replacing conventional filters in commercial building HVAC systems, thus easily retrofitted to existing facilities. Three different formulation avenues will be pursued: (1) gel application to a polymeric screen mesh, a screen made of the IQ-Gel, a commercially available filter, or a carbon cloth, such as ASZM-TEDA; (2) construction of a fiber bed of the gel; and (3) forming gel pellets or coating the gel on polymeric or carbon beads.

The tasks to be completed during this project are: (1) gather data on targeted CB threat agents and other toxic/hazardous chemical agents, develop a list of surrogate gaseous and particulate test compounds, and assess the vulnerabilities and capabilities of ASZM-TEDA and HEPA filters; (2) formulate the IQ-Gel into three formulations, validate the performance of each, and optimize; (3) validate the complete system under realistic test conditions; (4) prepare ten prototype commercial filters; and (6) develop description and drawings, model predictive performance,

and complete all required deliverables and reports. Included in the project is an optional task to develop a lifetime indicator interferometric sensor that will be imbedded into the IQ-Gel filter system to measure lifetime.

INTRODUCTION:



There is growing concern among civilian and military communities about the potential of terrorist attacks with chemical and biological (CB) warfare agents. The most indiscriminate delivery method of such agents is via an aerosol attack so that the agents are transported to the objective as gases or aerosol mists, in which particle or droplet sizes are in the respirable size range (≤0.6 μm), and so that a sufficient dose of the CB agent is transmitted to cause sickness and/or death. A particularly vulnerable area for attack is building ventilation (HVAC) systems at the outdoor air intake. An HVAC system is designed to draw in large amounts of outside air, mix it with a percentage of air already in the building, and transport that mixed air throughout the building, particularly to occupied zones. The ventilation system is the air pathway throughout a building. Release of a CB agent near a building's outside air intakes will allow distribution of the agent throughout the building, unless some type of filtration/air cleaning system exists in the ventilation system that captures the agent preventing agent transference into the building air and ventilation system. *We are proposing to develop this type of filtration/air cleaning system.* Any building is vulnerable to this type of attack with a CB agent or other types of toxic/hazardous, more common and readily available chemicals (such as phosgene, chlorine, hydrogen sulfide, methyl mercaptan, or arsine). US embassies and military buildings potentially are targets of this type of threat.

POTENTIAL USERS

The proposed technology has dual use capabilities for both military and civilian applications. Potentially it could be used in any building HVAC system, since it should remove common airborne pollutants, as well as CB

agents. US Embassy buildings, military facilities, commercial buildings, and personal residences are potential for use areas for the proposed filtration/air-cleaning/capture system. The proposed system should replace the currently used particle filters in the building HVAC systems. The sensor as a filter lifetime indicator will have many applications beyond combining with the IQ-gel filter. Currently such an indicator of filter life, especially for gases removal, does not exist.

TECHNICAL APPROACH:

We are proposing to develop an enhanced filtration/air-cleaning/capture system that will remove and trap airborne chemicals and particles from an airstream. Our proposed system will use the GTRI- patented IQ-Gel (US Patent 5,529,609), removing gaseous agents by adsorption/absorption and particulate agents (including biological agents) by adhesion, and a sensor or optical system indicating filter exhaustion. The IQ-Gel will be formulated so it that can be placed into standard filter housings replacing conventional filters in commercial building HVAC systems, thus easily retrofitted to existing facilities. Three different formulation avenues will be pursued: (1) gel application to a polymeric screen mesh, a screen made of the IQ-Gel, a commercially available filter, or a carbon cloth, such as ASZM-TEDA; (2) construction of a fiber bed of the gel; and (3) forming gel pellets or coating the gel on polymeric or carbon beads. Sensors or reactive light sources will be incorporated in the filter system at multiple points to indicate the progression of a threat agent through the filter system bed to prevent exhaustion of the filter system and breakthrough of the agent into the building supply air. The optimal criteria to be met by the IQ-Gel filter system are: 2'x2'x12" filter size, pressure drop across the filter system $\leq$3.0 iwg, pressure drop across each individual filter section $\leq$1.5 iwg, particle removal efficiency >99.97% for particle sizes between 0.3-10 μm, adsorption efficiency >99% for nerve, blister, and blood agents from ambient concentration of 5X IDLH with a breakthrough time of $\geq$15 minutes, and broad adsorption spectrum for acid-forming and basic toxic chemicals. Specific goals to be pursued are: a filter lifetime of three years, low cost, regeneration of the filter rather than disposal, and the development of a sensor for filter lifetime indication.

In conjunction with the IQ-Gel filter system, an indicator/lifetime/breakthrough sensor system is proposed as Option 1, which will be imbedded in several points of the filter media to measure the progression of a threat agent into the filter media. The sensors will detect potential failure or actual failure of the filtering ability of the IQ-Gel filter system. The sensor should be able to determine the identity of the breakthrough analyte and provide a warning to change the filter, shut down the filtering system or evacuate the building. This sensor will be based on the GTRI-

patented interferometric sensor (US patent 4,940,328), which has been used for sensitive detection and quantitation of a number of chemical and biological species. This sensor consists of an optical planar waveguide interferometer, which contains thirteen sensing regions on a single miniature platform.

The IQ-Gel

The IQ-Gel matrix consists of an acrylamide polymerization with the cross-linking agent, n-methylol acrylamide, in the presence of a plasticizing agent, glycerol. The IQ-Gel's properties of tack, viscosity, tensile strength, and pliability can be adjusted by varying the ratios of the ingredients and by varying reaction time between the ingredients, which will allow the formulation of the gel into the proposed three filter forms. Particles adhere to the media for removal from the air stream, and after adhesion move into the interior of the gel mat. The adhesion is dependent on surface energy rather than particle size, therefore the IQ-Gel will remove large and small particles, including living organisms – dust mites, bacteria, viruses, and fungi. Once a particle has adhered, it is not released back into the air stream. Gaseous pollutants are removed by a combination of adsorption and absorption. The non-optimized prototype filter, developed at GTRI during previous research projects, has demonstrated a capacity ratio for volatile organic compounds (VOCs) greater than seven times its weight, based on challenge mass and loading weight of challenge gas mixtures of authentic standards. This is capacity is significantly greater than most other known adsorbents. No re-emission was detected. Additionally the IQ-Gel does not support microbiological growth, since it does not have sufficient water activity to allow for proliferation and amplification of the organisms. Additional materials can be added to the IQ-Gel to augment its performance, including dyes, absorbents or strengthening material. Adsorbents, like Zeolites, and reactive compounds, such as metal catalysts, can be incorporated to tailor its removal abilities to specific applications. The IQ-Gel is the only known air cleaning media that can remove gases, particles, and microbes in a single pass on a single media.

Description of Relevant Previous Work

IQ-Gel: The IQ-Gel prototype development was funded under two projects: (1) by S.C. Johnson and Sons and GTRI (total funding $50,000) resulting in the GTRI patent "Air Cleaner Having a Three Dimensional Visco-Elastic Matrix of Material" (Patent #5,529,609) (report submitted to S.C. Johnson and Sons 1995), and the Advance Technology Development Center (ATDC) under the Faculty Research Commerialization Program (total funding $54,000) (report submitted to ATDC 1998). From this work, prototypes of IQ-Gel filter system have removed 99% of particles and an average of greater than 90% of organic compound. The drawback to

commercialization so far has been formulating the IQ-Gel into a form with sufficient inflexibility to be used in an HVAC system. The two previous projects were not sufficiently funded to be able to carry out this task. The results of these projects have not yet been published in peer-reviewed literature, since the IQ-Gel is still considered to be proprietary to GTRI, and we have chosen to maintain that data within GTRI until the IQ-Gel is formulated into a form that meets the needs for commercialization and manufacture by an industry partner.

During the first project, jointly funded by GTRI and S.C. Johnson and Sons, the IQ-Gel was first made, a prototype stand-alone air cleaner was constructed, and the IQ-Gel was patented. During this project, the original formulation for the IQ-Gel was developed. The IQ-Gel was encased between two pieces of polypropylene mesh, and a small stand-alone air cleaner was designed and evaluated. A schematic of this air cleaner is shown in Figure 1. The air cleaner was installed in office to determine the change in airborne particulate levels and lifetime. The respirable-sized

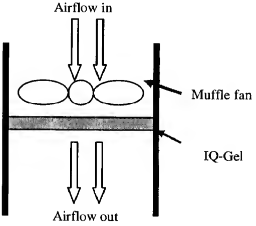


Figure 1. Schematic of prototype IQ-Gel stand-alone air cleaner.

particles (<1.0 μm) were reduced by approximately 50% for a 30-day time period. At the end of this time, the IQ-Gel, which had been colorless at the start of the experiment, was black and dirty in appearance. Another IQ-Gel filter was installed in the GTRI 28.3 m^3 dynamic environmental chamber (Figure 2). The IQ-Gel filter was challenged with ambient particles and a mixture of six organic gases representing different classes of organic compounds over a 30-day time period. The results of the organic challenge are shown in Table 1. The advantages of incorporation of adsorbents, such as Zeolite A, for enhanced VOC adsorption were investigated. Although enhanced removal efficiencies were obtained, the gel without added adsorbents performed sufficiently well for the project goals project without the added expense of the additional adsorbents. The IQ-Gel filter was challenge tested with *Aspergillus niger* at parts-per-

thousands levels for several days by Dr. Sidney Crow of Georgia State University, a expert in indoor microbial contamination. Dr. Crow found significant capturing of the microbial

IQ-Gel
air
cleaner

Figure 2. Prototype IQ-Gel air cleaner installed in GTRI 28.3 m^3 dynamic environmental chamber.

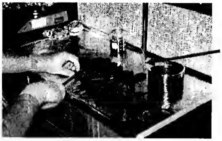
Table 1. VOC reduction % for challenge gas mixture.

Day #	% Reduction					
	Methylene Chloride	Ethyl Acetate	Methyl Ethyl Ketone	Trichloro-ethane	Hexane	Total Volatile Organic Compounds
1	62	47	59	40	79	59
2	85	55	74	44	81	73
4	95	70	84	86	90	92
5	97	80	80	85	93	93

particles by the IQ-Gel and detected no growth on the gel. Dr. Crow then placed the IQ-Gel filter into a microbially contaminated humidity chamber used to contaminate various materials and media and promote growth on these materials. The IQ-Gel remained in the chamber for 30 days. No growth on the IQ-Gel was detected. Dr. Crow determined that the IQ-Gel has very low water activity , and therefore could not support growth.

During the second project, the IQ-Gel was formulated into several different forms, by varying the reaction conditions and ingredient amounts, ranging from a pourable liquid to a diceable hard material (Figure 2a). The latex form of the gel was onto metal grease filters and residential fiberglass furnace filters (Figure 2b). Three grease trap filters with IQ-Gel latex were tested in the large chamber facility and at a Florida yacht club. The particle count readings are given in Table 2. The particle background levels in the chamber varied due to the outside air introduced into the chamber. The particle concentration varied due to the weather outside and the traffic near the intake. Each was effective in removal of particles by 22 hours; all three were removing approximately 20% of

particles in the 0.3-0.5 μm and 0.5-5.0 μm range. The poured filter #1 and sprayed filter #2 removed greater than 50% of the 5.0-10.0 μm



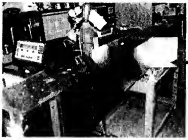


Figure 2. IQ-Gel formulation (a) allowing it to be cut into cubes and (b) coated on fiberglass.

range particles. Sprayed filter #1 only removed 6.65% of the 5.0-10.0 μm range particles, but this possibly may be due to the higher concentration of these particles to which the sprayed filter #1 was exposed. Using these two matrices for support of the gel did not allow for a sufficient amount of the gel to be coated on the matrix for lifetimes greater than a few days. The IQ-Gel-coated was coated on metal grease filters installed in place of high efficiency filters in the HVAC system of a Florida yacht club for 48 hours. During this time, occupants remarked about how clean and pure the air felt. Measurement of contaminants in the indoor air with the IQ-Gel filters in place indicating some improvement in the indoor air quality. Based on these results, we determined that the IQ-Gel captures greater than seven times its weight in airborne pollutants, but the lifetime was only a few days due to the small amount of material that could be sprayed onto the support media without damaging the support media. These filters were coated with approximately 63 g of the IQ-Gel. Based on the data, we calculated that at least three times this amount of IQ-Gel must be coated on the base media to achieve the desired lifetime of at least three months. We hypothesized that IQ-Gel formulation should be in a pellet or coated bed, a fiber bed, or coated on a media with matched surface energies. Dr. Gooch, one of the co-inventors, has demonstrated that the IQ-Gel can be extruded into fibers and formed into pellets. Three to five years lifetime has not been quantitatively tested, but has been anecdotally tested. Batches of the IQ-Gel have been in the GTRI laboratory for three to four years and have been noted to still function as air cleaning media. No data has been collected to determine the removal efficiency of the IQ-Gel under different climatic conditions, but since the IQ-Gel is greater than 90% water, and since it does dry-out during use, high humidities should increase the IQ-Gel effective lifetime.

Table 2. GTRI 28.3 m^3 chamber particle data during second research project.

Sample description	Average Particle counts							
Spray filter #1	**Particle size 0.3-0.5 µm**	**% removal**	**Particle size 0.5-5.0 µm**	**% removal**	**Particle size 5.0-10.0 µm**	**% removal**	**Particle size >10.0 µm**	**% removal**
Chamber blank, outside air	183,968		14,578		183		0	
Chamber blank, before filter	175,422		10,973		28		0	
Chamber blank, after filter	191,531	-9.18	13,948	-27.11	46	-66.37	0	0.00
2 hours, outside air	89,588		5,543		101		0	
2 hours, before filter	68,019		3,848		34		0	
2 hours, after filter	67,809	0.31	4,436	-15.26	104	-201.03	0	0.00
22 hours, outside air	54,446		4,087		53		0	
22 hours, before filter	102,801		9,493		218		0	
22 hours, after filter	83,113	19.15	7,632	19.61	203	6.65	0	0.00
Poured filter #1	**Average Particle counts**							
Chamber blank, outside air	322,592		30,986		9		0	
Chamber blank, before filter	205,577		20,144		70		0	
Chamber blank, after filter	196,127	4.60	18,090	10.20	14	79.72	0	0.00
2 hours, outside air	272,222		20,843		7		0	
2 hours, before filter	276,345		23,520		5		0	
2 hours, after filter	134,049	51.49	9,226	60.78	14	-166.27	0	0.00
22 hours, outside air	326,650		31,237		4		0	
22 hours, before filter	288,983		27,294		160		0	
22 hours, after filter	224,889	22.18	19,698	27.83	70	56.66	0	0.00
Sprayed filter #2	**Particle size 0.3-0.5 µm**	**% removal**	**Particle size 0.5-5.0 µm**	**% removal**	**Particle size 5.0-10.0 µm**	**% removal**	**Particle size >10.0 µm**	**% removal**
Chamber blank, outside air	81,504		6,500		6		0	
Chamber blank, before filter	163,742		13,386		21		0	
Chamber blank, after filter	150,866	7.86	13,192	1.45	11	46.73	0	0.00
2 hours, outside air	145,305		13,740		10		0	
2 hours, before filter	132,147		12,672		21		0	
2 hours, after filter	101,416	23.26	9,290	26.69	10	50.79	0	0.00
22 hours, outside air	147,158		16,332		47		0	
22 hours, before filter	120,184		14,207		66		0	
22 hours, after filter	91,417	23.94	11,370	19.97	24	64.51	0	0.00

"PROJECT ATLANTA" CERTIP AGENT DATABASE: The GTRI Center for Emergency Response Technology, Instruction and Policy (CERTIP) was formed to address the increasing risk of terrorist use of chemical and biological weapons of mass destruction and the increasing risk of inadvertent release of such agents. The CERTIP vision is that new technologies and adaptations of existing technologies are needed to support the six communities which must respond to such incidents: law enforcement, intelligence, hazardous materials, military, medical and emergency management. The two-year mission of CERTIP is to create a national public-private partnership that will support research and development of needed technologies. There are over 40 CERTIP partners, predominantly in the Southeast, including university, civilian and military federal agencies, and Georgia agencies.

The CERTIP program, "Project Atlanta", sponsored by the US Marine Corps Warfighting Laboratory, seeks to develop affordable sensor, information technology, reach back, and triage and decision aid technologies. As part of this effort, a database of agents was developed, which includes chemical and biological warfare agents and hazardous chemicals that are likely candidates for use by terrorists. This CERTIP database includes chemical and physical characteristics as well as human symptoms resulting from exposure, and contains data for the chemical agents and toxic industrial chemicals, including medical information (median lethal dosage, median incapacitating dosage, time of onset of symptoms, persistence, mechanism of action, treatment, rate of detoxification, and symptoms), chemical information (molecular weight, chemical state at 20C, physical characteristics, vapor density, liquid density, freezing/melting point, boiling point, flash point, decomposition temperature, stability, heat of vaporization, vapor toxicity, and volatility), personal protective equipment required, decontamination procedures, means of detection, and alternate uses of chemical. The collected data is being used for the formation of a decision matrix which will allow for the preliminary identification of chemicals based upon "observables" such as: pupil size, pupil photosensitivity, skin color, sweating, twitching, coughing, choking, skin pain, respiration rate, bronchial spasms, excretions, diarrhea, hallucinations, sneezing, and others.

The CERTIP Agent Database (CAD) is being used to structure the development of the technologies to be demonstrated in Project Atlanta: (1) interferometric sensors tuned to appropriate agents for use in hand-held devices or devices used to protect buildings; (2) CERTIP "Palm pilot" Decision Aid (CPPDA) for first responders, which will allow a first responder to rapidly identify the class of agent, and lead the first responder into a database of

information including a standardized set of information relating to the identified chemical; (3) a network providing wireless reach-back to medical authorities anywhere in the world, including data on agent dispersal, decontamination procedures and detailed agent characteristics; and (4) a radar "flashlight" device to detect the movement of casualties or terrorists through walls and doors. CERTIP plans to conduct limited demonstrations of these technologies during the next two years, followed by use of the technologies in a large-scale exercise in FY02.

Interferometric Sensor (Proposed as Option 1): The proposed interferometric lifetime sensor is based on the GTRI-patented interferometric sensor, which has been used for sensitive detection and quantitation of a number of chemical and biological species, and which is being used for "Project Atlanta". This sensor consists of an optical planar waveguide interferometer, which contains thirteen sensing regions on a single miniature platform. Planar waveguides have evanescent fields sensitive to index of refraction changes in the volume immediately above the waveguide surface. These fields extend up to 5000 Å above the surface. Placing a chemically sensitive film within this region provides the basis for a chemical sensor (Figure 3). Polymer/analyte interactions change the index of refraction causing the propagating light speed, or phase, to change in a direction opposite to that of the index change. To measure this change a reference-propagating beam, adjacent to the sensing beam, is optically combined

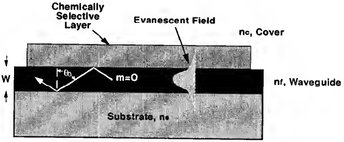


Figure 3. Diagram for single mode waveguide with buried evanescent field.

with the sensing beam creating an interference pattern of alternating dark and light fringes (Figure 4). When

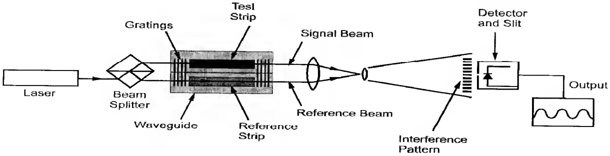


Figure 4. Diagram of non-integrated waveguide interferometer setup.

chemical or physical changes occur in the sensing arm, the interference pattern will shift producing a sinusoidal output. With detectable sensitivities on the order of 0.02 π radians, index changes less than 10^{-6} can be measured[3,4]. Tailored chemistries applied to the waveguide can be passive (e.g.; inducing swelling or dissolution in a film) or active (e.g.; containing reactive or binding sites). Fast and reversible chemistries usually are the goal, in most cases, for both gaseous and liquid applications; however, an integrating chemistry can just as easily be designed to determine very low concentrations of a given analyte such as chemical warfare agents. Passive mechanisms are used when the target analyte is relatively inert, *i.e.* aromatic and chlorinated hydrocarbons. Active chemistries developed include tailoring the acid-base strength of the sensing film, the nucleophilicity or electrophilicity of the film, and antibody-antigen binding. Sensitivities range from the low ppm to low ppb for vapor and aqueous applications, and 0.01 pH units or ng/mL for biologicals. It also is possible with the multiple sensors channels on a in single platform, that a combination of active and passive chemistries can be used together, thereby tailoring the sensor to meet specific needs. The currently developed sensor platform has 13 interferometers on a 1x2-cm chip (Figure 6). All components needed to launch the optical beam, interfere the optical waves and couple out the interference pattern are fabricated into the waveguide chip. A laser illuminates all channels across the width of the

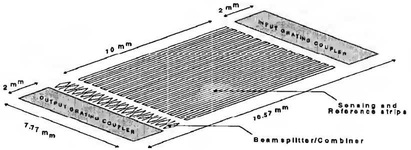


Figure 6. Integrated chip with 13 interferometers, gratings and optics.

input grating. A patterned layer of thick SiO_2 defines the channel lengths. In the case of the passive organic solvent sensor, coarse, medium and fine channels allow for increased sensitivity and dynamic range. Four polymers (three using the dissolution mechanism and one polymer using free volume filling) are used to provide a patterned output for analyte identification and concentration determination. In the active sensor, coarse and fine channels have been used in addition to separate channels for canceling possible interferants. Field testable units have been assembled with onboard electronics capable of converting interferometric signals to total phase shift, and reporting concentration and identification of single analytes and mixtures through pattern recognition and specific reactions. Vapor phase units are approximately 2" x 3" x 6" in dimension. Further reduction in size is expected in the near future.

The sensor is being applied to a number of environmental, industrial, and packaging detection problems for a number of government (military and civilian) and industrial sponsors. Several of the applications to which the GTRI sensor is being applied and/or developed are: BETX sensing in water, humidity measurement, ammonia sensing during fertilizer application, airborne influenza virus, *salmonella* on processed poultry, *E-coli* detection, toxic industrial chemicals, and CB agents. For example, by incorporating acid-base chemistry into the polymer, a pH sensor and an ammonia sensor have been developed. The response of these sensors is fast and reversible, since they use an active chemistry. The sensitivies for ammonia are in the <100-ppbv range. The ammonia sensor uses two different pKa's. One arm of the interferometer has a pKa, which lies above that of ammonia and the other arm a pKa, which lies below that of ammonia. Ammonia diffuses equally into both arms equally, but reacts with one to produce dipoles. For example, citric acid was chosen to react with ammonia. Citric acid has three protons with pKa's of 3.1, 4.8 and 6.4. The proton with the pKa of 6.4 is the most useful proton to reversibly react with ammonia. The other two are too acidic to be reversible and will be tied up in a reaction with a polymer adjusted to the appropriate pH (Figure 7)[7].

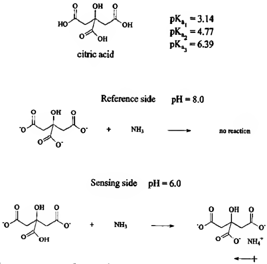


Figure 7. Citric acid's pKa's and structure and reactions at pH 6 and 8.

A solution of a basic polymer, polyethyleneimine-80% ethoxylated , pH 11, is titrated with citric acid. The polymer tethers the citric acid, holding it within the evanescent field. The reference arm contains polyethyleneimine-80% ethoxylated titrated to pH 8.0 so that no acid protons are left on the citric acid. Only the much higher pKa protons on the nitrogen of the polyethyleneimine remain. The sensing arm is titrated with citric acid to pH 6.0 leaving one proton on each citric acid capable of reacting with the ammonia. Polymer films are 5000Å thick, burying the evanescent field. Ammonia reacts reversibly with the remaining citric acid protons. Sensitivities are in the sub 100-

ppbv range and show that active chemistries provide substantial improvement over passive chemistries (Figures 8 and 9)[5,7].

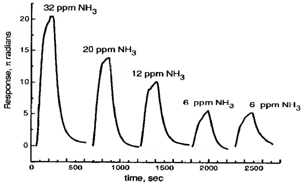


Figure 8. Time response to ammonia at various concentrations.

The interferometric setup has been condensed so that all optical components are integrated on a single substrate (Figure 10)[7]. Only the laser and detector remain external. The laser beam is coupled into the waveguide by use of a grating coupler. The channels are not defined in the Si3N4 waveguide nor are they defined by separate

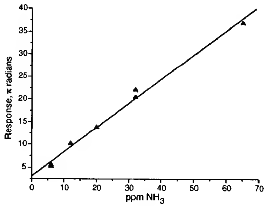


Figure 9. Linearity of ammonia response.

laser beams but by the deposited chemistries. After the beam passes under the chemistry, the two sides of the beam are guided by total internal reflectors (TIR) to a beam splitter, which combines the beams to form the interference pattern. The interference-patterned light is reflected off the backside of the TIRs and directed to an output grating. The output grating then directs the light to the detector.

PROJECT OUTLINE

STATEMENT OF WORK

During this proposed research project, an enhanced filtration/air-cleaning system using the IQ-Gel will be developed that will remove and trap hazardous, toxic chemical and biological agents from an air stream. The IQ-Gel will be formulated into the optimum form to perform this function for a minimum of three to five years. Different formulations to be pursued include (1) gel application to a polymeric screen mesh, a screen made of the IQ-Gel, a commercially available filter, or a carbon cloth, such as ASZM-TEDA; (2) construction of a fiber bed of the gel; and (3) forming gel pellets or coating the gel on polymeric or carbon beads. The IQ-Gel system will be evaluated in the GTRI 28.3 m^3 dynamic environmental chamber and an ASHRAE 52.2 test rig. During the evaluation stage, the optimal location of the filter in the HVAC system will be determined. It is expected that the filter should replace the conventional filters already installed in an HVAC system, upstream of the cooling coil and downstream of the outdoor air supply. Combination of the IQ-Gel with currently available technology such as HEPA filtration or activated carbon (specially ASTM-TEDA) filters will be considered. An optional lifetime sensor is proposed to be developed that will be imbedded in the filter to indicate exposure and lifetime of the filter. The sensor is proposed in as two options. One sensor only will measure filter lifetime. The second option would detect the presence of an agent. Ideally this sensor also would close the outside air dampers whenever an attack is detected preventing additional exposure to the agent. If Option 1 is not funded, alternate means of determining filter lifetime will be investigated. Proposed solutions include, investigation of the use of other sensors that have been developed as lifetime indicators or incorporation of a chemical that would react as a chemiluminscence or fluorescence agent that can be detected by currently available sensors. A predictive performance model will be developed for the IQ-Gel system. Ten prototype filters will be delivered for governmental evaluation before the end of the project.

Task 1: *Gather data on targeted chemical/biological threat agents and other toxic/hazardous chemical agents that could pose a threat and develop a list of surrogate gaseous and particulate test materials. Evaluate targeted CB threat agents for adsorption/filtration requirements to be meet for required efficiency specifications. Assess vulnerabilities of currently available ASZM-TEDA activated carbon and HEPA filters; assessing how the IQ-Gel can reduce these vulnerabilities and replace or combine with these technologies retaining the chemisorption properties of ASTM-TEDA activated carbon.* Many candidate threat agents have similar structures and properties to readily available, less toxic industrial chemicals. For example many nerve agent chemicals are organophosphate based, the same

chemistry as used for many pesticides; therefore organophosphate pesticides may be substituted as a surrogate test material for nerve agents. During this task, threat agents of interest will be classified and appropriate surrogate, less toxic chemicals (volatile, semi-volatile, and particulate) will be selected for challenge testing of the IQ-Gel filter to determine efficiency, performance, and lifetime. GTRI database of CB threat agents developed under "Project Atlanta will be used as the basis to select appropriate surrogate chemicals. The database will be shared with the government technical supervisors of this proposed project to determine the necessity for increasing the threat agent data in the existing GTRI database, based on the agents of interest to this project. TSWG should share with GTRI the Hazardous Materials Database, the Detector Simulant Kit, and the Detector Evaluation data that have been developed as TSWG funded projects, or at least the relevant information from these projects. These will be assessed for their applicability to this project.

ASZM-TEDA activated carbon has been formulated to have specific chemisorption properties for chemical threat agents in military personnel respirators, but has not been applied to commercial building filtration. Carbon filters traditionally have been used for gaseous compound filtration in respirators, clean rooms, commercial buildings, and stand-alone air cleaners. These filters have not been adequately tested to determine capture efficiency, efficacy, retention of adsorbed compounds over time and under various conditions, lifetime, breakthrough, etc. Although these filters are widely used, a major issue of use is when to change the filter. The GTRI researchers proposing this project currently are funded by the Association of Heating, Refrigerating, and Air-Conditioning Engineers (ASHRAE) to develop field test methods to measure the contaminant removal effectiveness of gas phase air filtration equipment. During this project, the researchers are establishing methodologies to measure and provide data about contaminant removal effectiveness of the gaseous air filters in commercial buildings. These methods need to be developed before data shortages to determine capture efficiency, efficacy, adsorbed compounds retention, lifetime, etc. can be solved. The methods and data developed during this project will be applied during this task to assess the ASZM-TEDA vulnerabilities based on available literature. The government will provide ASZM-TEDA filter media to the GTRI researchers for evaluation.

Task 2: *Formulate GTRI-patented IQ-Gel into the three forms, validate its performance, and optimize for maximum efficiency and lifetime for targeted applications.* The criteria for filter formulation evaluation are: 1) sufficient airflow through the filter, 2) pressure drop across filter system, 3) stability of gel on support matrix, 4) removal and capture efficiency for targeted particles and aerosols, gases, and microbes 5) filter capacity, 6) filter lifetime, and 7) potential for commercialization. A critical point for making an IQ-Gel filter with sufficient quantities of gel to reach

desired lifetimes is surface matching of the gel with the appropriate support matrix, and for the surface matrix to have enough rigidity to hold the weight of the gel. An approach to this problem is the selection of a polymeric screen with the appropriate surface characteristics. Dr. Campbell will investigate this approach, since this is similar to the problems that he solved in the development of waveguides for the interferometric sensors. The proposed filter system may be used in conjunction with an existing pre-filter in the HVAC system to remove the larger particles to increase the gel filter lifetime and may be used in conjunction with appropriate carbon or impregnated filters. It is predicted that the three formulations will be made and preliminarily tested. The formulation that best meets the performance criteria will be selected to continue with validation with the surrogate compounds selected in Task 1. An appropriate filter housing system will be designed meeting the criteria that the filter system replace currently used HVAC filters. (This will be done with Mr. H.E. Burroughs, PE of Building Wellness Consultancy, an internationally recognized expert in filtration and indoor air quality). Validation will be with modified ASHRAE 52.2 Standard test method and using the method specified test rig modified for gaseous compound challenge testing, as well as particle efficiency testing. The particulate removal and capture efficiency will be measured by challenging the gel-filter with a range of dry KCl particles sizes. (ASHRAE 52.2 uses a particle size range of 0.30 to 10.00 μm, but this will be extended to 0.10 to 500 μm for this project.) The removal efficiency is calculated by measuring the concentration differences up- and downstream of the test filter. Organic (volatile and semi-volatile) compounds of interest will be injected into the modified ASHRAE 52.2 test rig and the removal efficiency calculated by measuring the difference in concentrations up- and downstream of the test filter. Lifetime will be evaluated by challenging the gel-filter with extremely high concentrations of target compounds to measure the capacity of the IQ-Gel for these substances, both gaseous and particulate. The lifetime will be predicted from these data, similar to ASHRAE Draft Standard SPC145. GTRI is currently funded by ASHRAE for a research project on gaseous filtration efficiency and service life determination that will provide input into the filter testing methods proposed for this project.

Materials incorporation into the IQ-Gel will be investigated to obtain greater lifetime, overall removal/capture efficiency, and/or specificity of removal/capture efficiency for target species. These materials may include adsorbents, such as Zeolites, metal salts, or other reactive chemicals. Reactive chemical incorporation may be particularly useful for the capture and degradation of nerve and blister agents of acid-forming chemicals. Coating of the filter on ASTZ-TEDA cloth or particles, or incorporating the particles into the IQ-Gel may be pursued.

Lifetime indication methods will be investigated, particularly if Option 1 is not funded. An alternate sensor that can be imbedded in the filter will be sought, such as an NDIR sensor. The government will provide the GTRI researchers data and information about the sensors that have been developed for detection of CB agents to determine if any of these will meet the needs to indicate the need to change the IQ-Gel filter. The government will supply the sensor if chosen. Ideally the selected filter will be able to signal the outside air dampers on a building to close due to the presence of an external attack. Alternatively we will investigate the incorporation of reactive compounds into the IQ-Gel that will react with CB and toxic airborne agents resulting in a chemiluminescence or fluorescent reaction that can be detected by available detectors. These reactions should be concentration dependent so that a concentration close to the lifetime of the filter must be reached before the chemiluminescence or fluorescence occurs. The government will provide the GTRI researchers data and information about chemiluminescent or fluorescent detectors that have been developed for the detection of CB agents. The government will supply the sensor if chosen.

Task 3: *Validate complete system in GTRI 28.3 m^3 environmental chamber system and in an ASHRAE 52.2 test rig* with surrogate compounds and particles. Installation of the IQ-Gel filter in the HVAC system of the chamber will allow us to simulate its use in a building. The effects of changing temperature and humidity and challenge mixtures on performance also will be determined using the testing procedure described above. The performance under changing environmental conditions and differing pollutant loadings is a critical performance criterion. We predict that the IQ-Gel filter will perform best under higher relative humidity conditions, because the prototypes appeared to be very dry (no longer moist) at failure. Lifecycle calculations will be based on the cost of materials and construction, the efficacy and efficiency, any increased energy costs or equipment costs for operation, and potential costs for disposal or renewal (if possible). Challenge testing will be based on the selection of surrogate and toxic chemicals from Task 1 and in conjunction with the governmental technical advisors. At a minimum, cigarette smoke and a VOC mixture containing toluene, formaldehyde, heptane, ethylbenzene, methyl ethyl ketone, and methylene chloride will be used.

Storage and shipping conditions also will be determined and evaluated during this task. At a minimum the filter will have to be protected from passively adsorbing large amounts of chemicals during storage and shipping, thus shortening the filter's lifetime while installed.

Task 4: *Prepare ten commercial-type 2' x 2' x 1' filters for government evaluation.* These will be done in conjunction with the manufacturing industry partner, expected to be Ecolab Inc. The filters will be made using the methods developed in Task 2. The cost of the filter also will be determined during this task. The cost will be estimated for a single filter and larger quantities of the filter, i.e. 50, 100, or 1000 filters. It is not possible to estimate the cost of the final filter until the formulation has been determined.

Task 5: *Develop description and drawings of adsorption/particle filter system in typical building system, specifications, model to predict system performance in buildings, and users' manual. Included in this task are the required reports and deliverables outlined in the deliverables section of this proposal.* The final designs and report on performance, installation, performance, etc. will be prepared both on electronic and hard media. Dr. Kaiss Al-Ahmady of Indoor Air Solutions, a recognized expert in indoor air modeling will prepare a model to predict system performance in buildings.

Option 1: *Develop lifetime indicator sensor. Incorporate into filter system and validate performance with surrogate compounds.* The interferometric sensor will be developed for the selected targeted compounds. It is anticipated that the sensor chemistry will be developed to measure compound classes rather than specific compounds to increase the likelihood of detection. Particles will be detected by changes in optical density on the surface. Two options exist for the deployment of this sensor: the sensor can be incorporated into the IQ-Gel filter itself or placed downstream from the filter. During its effective lifetime, the filter may be challenged potentially with many different chemical species, even if it is never exposed to CB agents.. It is hypothesized that when failure occurs the last chemical species encountered by the filter will be the breakthrough compound, never being adsorbed by the filter. This simplifies the sensor's design. It takes two separate interferometers with different indexed polymers to distinguish the difference between a small amount of an analyte with a large polymer/analyte index difference and a large amount of analyte with a small polymer/analyte index difference. But also by taking the ratio of the phase change between two different interferometers, a value is obtained that is unique to every single compound since each compound has a unique index of refraction. By having at least two interferometers with different polymers, identification of the breakthrough chemical compound is possible by having a "look-up table" of compounds and their refractive indices either in an accompanying computer or stored on-board the sensor.

Two interferometers are needed at the very least for this sensor application but it would be more advantageous to utilize more of the sensor's real estate. Additional interferometers with different polymers provide

additional verification and compensation for any humidity effects as well as mechanical and thermal noise. Initially, starting with four polymers and four or eight interferometers (providing for coarse and fine sensors, respectively, if needed), enough information should be obtained to sense the breakthrough and identity of an analyte. It should also be able to handle simple mixtures if the breakthrough hypothesis is not correct, compensate for humidity changes and monitor the stability of the sensor, laser and detector. Presently, the number of interferometers on a single chip stands at thirteen. However, this number was dictated by the detector array available at the time of the initial design, and recent improvements in detector technology allows up to fifty interferometers on the same chip.

There are two possible ways to wed the sensor technology with the IQ-Gel filter in order to provide information as to the adsorbing status of the IQ-Gel. The first is to embed the sensor directly into the IQ-Gel matrix preferably near the trailing end of the filter. As the filter charges with contaminants, the wavefront of the contaminates eventually will reach the sensor, and the sensor will respond prior to breakthrough. The sensor can be configured to record data continuously or intermittently. Having a sensor embedded in the filter obviously requires at least one sensor per each filter. In the second possible configuration, one sensor can be used to evaluate the effectiveness of several filters. In this scenario, a sensor is constructed as a stand-alone device. This device either is placed in the stream down wind from the filter or the sensor has a sampling tube that draws air from behind the filter and over the sensor. However, the only way to test the effectiveness of the filter since one can not be sure at the time of sampling that the filter is being challenged is to challenge it with a known amount of analyte. An aliquot of a substance with a known sensor response is injected into the air stream upline from the filter. The sensor is sampling downstream from the filter. The amount of response at the sensor indicates the effectiveness of the IQ-Gel filter. With a stand-alone sensor, numerous filters can be evaluated in the course of a day. However, breakthrough can happen at any time between sampling so one will only know that failure occurred sometime between successive samples. One way to insure that the contaminant does not get passed along between sampling is to have two filter in series and the challenged sampling takes place between the two filters. This way when the first filter fails between testing the second serves as backup. If sampling indicates failure of the first filter, it is changed out leaving the second to continue serving as backup.

The cost of the sensor, both singly and in mass quantities will be calculated once the design of the final sensor is determined. In general the interferometric sensors developed by GTRI are costing between several tens of dollars to several hundreds of dollars depending on the application and sophistication.